NORTH ATLANTIC TREATY ORGANIZATION

ORGANISATION DU TRAITE DE L'ATLANTIQUE NORD

MILITARY AGENCY FOR STANDARDIZATION (MAS)
BUREAU MILITAIRE DE STANDARDISATION (BMS)

1110 BRUSSELS

MAS/188-MMS/4178
6 August 1991

To : See MAS Distribution List No. 2

Subject : STANAG 4178 MMS (EDITION 1) - TEST PROCEDURES FOR ASSESSING THE QUALITY OF DELIVERIES OF NITROCELLULOSE FROM ONE NATO NATION TO ANOTHER

Reference : AC/310-D/53 dated 4 April 1986

Enclosure : STANAG 4178 (Edition 1)

1. The enclosed NATO Standardization Agreement which has been ratified by nations as reflected in page iii is promulgated herewith.

2. The reference listed above is to be destroyed in accordance with local document destruction procedures.

3. AAP-4 should be amended to reflect the latest status of the STANAG.

ACTION BY NATIONAL STAFFS

4. National staffs are requested to examine page iii of the STANAG and if they have not already done so, to advise the Defence Support Division, IS, through their national delegation as appropriate of their intention regarding its ratification and implementation.

E. STAI
Major-General, NOAF
Chairman, MAS

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MAS188A

STANAG 4178
(Edition 1)

NORTH ATLANTIC TREATY ORGANIZATION
(NATO)

MILITARY AGENCY FOR STANDARDIZATION
(MAS)

STANDARDIZATION AGREEMENT

SUBJECT: TEST PROCEDURES FOR ASSESSING THE QUALITY OF DELIVERIES OF NITROCELLULOSE FROM ONE NATO NATION TO ANOTHER

Promulgated on 6 August 1991

E. STAI
Major-General, NOAF
Chairman, MAS

STANAG 4178
(Edition 1)

RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTES

AGREEMENT

1. This NATO Standardization Agreement (STANAG) is promulgated by the Chairman MAS under the authority vested in him by the NATO Military Committee.

2. No departure may be made from the agreement without consultation with the tasking authority. Nations may propose changes at any time to the tasking authority where they will be processed in the same manner as the original agreement.

3. Ratifying nations have agreed that national orders, manuals and instructions implementing this STANAG will include a reference to the STANAG number for purposes of identification.

DEFINITIONS

4. Ratification is "The declaration by which a nation formally accepts the content of this Standardization Agreement".

5. Implementation is "The fulfilment by a nation of its obligations under this Standardization Agreement".

6. Reservation is "The stated qualification by a nation which describes that part of this Standardization Agreement which it cannot implement or can implement only with limitations".

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

7. Page iii gives the details of ratification and implementation of this agreement. If no details are shown it signifies that the nation has not yet notified the tasking authority of its intentions. Page iv (and subsequent) gives details of reservations and proprietary rights that have been stated.

Agreed English/French Texts

STANAG 4178
(Edition 1)

NAVY/ARMY/AIR

NATO STANDARDIZATION AGREEMENT (STANAG)

TEST PROCEDURES FOR ASSESSING THE QUALITY OF DELIVERIES OF NITROCELLULOSE FROM ONE NATO NATION TO ANOTHER

Annexes: A. Test Procedures
B. Test Report Sheet

Related Document: None

AIM

1. The aim of this agreement is to standardize methods of test for assessing the quality for nitrocellulose used in the manufacture of explosives and propellants. The nitrocellulose is prepared from cellulose derived from cotton or wood, the cotton maybe in the form of waste or linters and the wood may be in the form of paper or pulp. The addition of stabilising agents to the nitro-cellulose is not permitted without the prior authority of the purchasing agency.

2. The procedures described in Annex A were developed to facilitate cross procurement and provide means by which countries can satisfy themselves that nitrocellulose received from abroad has been tested by acceptable means. They are not intended for use for quality control purposes during the manufacture of nitrocellulose.

3. This agreement is intended for use by the NATO Armed Forces.

AGREEMENT

4. The NATO Armed Forces of the ratifying nations agree to use the test procedures described in Annex A for assessing the quality of nitrocellulose when requested by the procuring nation.

IMPLEMENTATION OF THE AGREEMENT

5. This STANAG is implemented when ratifying nations agree to accept from other countries nitrocellulose when tested according to the procedures described in this STANAG.

TEST PROCEDURES

EXAMINATION OF NITROCELLULOSE

DESCRIPTION

The nitrocellulose used in the manufacture of explosives and propellants for Service purposes is prepared from cellulose derived from either cotton or wood. The cotton may be in the form of waste or linters and the wood in the form of paper or pulp. The nitrocellulose shall not have been deliberately stabilised by the addition of stabilising agents without prior permission of the purchasing agency.

TESTS TO BE APPLIED

(1) Preparation of Sample and Visual examination

(2) Determination of mineral matter

(3) Determination of grit

(4) Determination of organic matter soluble in ether/alcohol mixture

(5) Determination of organic matter insoluble in acetone

(6) Determination of fibre size/length by sedimentation

(7) Viscosity

(8) Determination of nitrogen content

(9) Determination of stability by the 132°C stability test

(10) Determination of stability by the heat test

(11) Examination for the presence of stabilising agents.

ANNEX A to
STANAG 4178

PRECAUTIONS - GENERAL

Dry nitrocellulose, particularly when in the form of dust, presents a serious fire and explosion risk and all necessary precautions on this account must be taken. Preparation of samples must be carried out in a flame-free room and care exercised in the transference of samples from one place to another in the laboratory.

Safety goggles or a face shield should be worn for all operations involving dry nitrocellulose.

Mercury or acid spills should be cleared away immediately.

1. PREPARATION OF SAMPLE AND VISUAL EXAMINATION

a. Nitrocellulose may be sampled at various stages of its manufacture. The samples are taken wet and require preliminary treatment before analysis can begin. Preparation of samples must be carried out in a flame-free room.

b. Place the material (approximately 150 g) between layers of thick filter paper which must be pure and free from chemical impurities and put it under a hand screw press (see NOTE 1 below). Subject it to tolerably severe pressure for 3 minutes. Then tease out loosely with the fingers and spread the nitrocellulose on a paper or aluminium tray (approximately 22.5 × 12.5 cm). Dry in an oven for 4 hours at 70°C. The oven should be water-jacketed and fitted with air ducts to provide efficient ventilation and should preferably be thermostatically controlled. Rub the dried sample out as finely as possible breaking down any lumps. Examine the material for presence of foreign matter and finally transfer it to a rubber stoppered bottle.

NOTE 1: It may be necessary to do a repeat pressing, after renewing the filter paper.

NOTE 2: Alcohol wet nitrocellulose shall be dried for 16 hours at 50°C and then one hour at 100°C.

NOTE 3: Sample preparation prior to determining the Heat Test shall be carried out as described in paragraph 10.5.

DISPOSAL OF WASTE MATERIAL

Place sample residues and material which has been used in the determination of the fibre size/length test in a wet waste pot for subsequent disposal.

Transfer the solvent solutions of nitrocellulose to a waste solvent container for subsequent burning.

REAGENTS - GENERAL

Unless there is an instruction to the contrary, the use of chemicals of Analytical Reagent quality is to be understood. If water is used as a reagent, it shall be freshly distilled or freshly boiled and cooled distilled water (or deionised water of equivalent purity).

2. DETERMINATION OF MINERAL MATTER

2.1 Outline of Method

Mineral matter is defined as the residue remaining after ignition of the nitrocellulose and calcination at 600°C.

NOTE: Certain specifications require the mineral matter to be carbonated; in such cases the residue left after ignition is treated with ammonium carbonate and dried to constant weight at 105-110°C, (see paragraph 2.2b. below).

2.2 Procedure

a. Transfer 2 ± 0.01 g of the sample to a tared platinum basin (weight W_1). Saturate the material with liquid paraffin (or other suitable material). Heat the mixture gently until it ignites. Allow it to burn away without further heating. Burn off any carbonaceous matter using a bunsen burner and complete the ignition in a muffle furnace at 600°C. Transfer the platinum basin to a dessicator, allow to cool and weigh (weight W_2). Carry out a blank determination under identical conditions on an equal volume of liquid paraffin (blank weight W_3).

b. If carbonation is required, wet the residue from 2.2 with 2 cm^3 of 5 per cent (w/v) ammonium carbonate solution. Place the basin on top of a 100°C oven until the residue is dry and then heat to constant weight. Repeat the carbonation and drying until the residue is constant in weight (W_4). Carry out a blank determination using the appropriate volumes of liquid paraffin and ammonium carbonate solution. (Blank weight W_5).

2.3 Calculation and Reporting

Calculate the mineral matter content and report as a percentage by weight of the sample as follows:

(a) Percentage mineral matter = $(W_2 - W_1 - W_3) \times 50$.

or

(b) Percentage mineral matter (carbonated) = $(W_4 - W_1 - W_5) \times 50$.

3. DETERMINATION OF GRIT

3.1 Definition

For the purpose of this test grit is defined as the amount of mineral matter, insoluble in hot concentrated hydrochloric acid and hot concentrated sodium hydroxide, which will not pass a No. 60 British Standard sieve or equivalent.

3.2 Apparatus

Two-inch diameter sieves having mesh sizes of 36 and 60 complying with British Standard 410, 'Test sieves' (or equivalent).

3.3 Procedure

a. Digest the residue from the determination of mineral matter (paragraph 2.2) with concentrated hydrochloric acid on a boiling water-bath. Allow the insoluble matter to settle, dilute the acid with an equal volume of water and decant through a 9 cm No. 41 Whatman filter paper or equivalent. Wash the insoluble matter on to the paper and wash the basin and filter paper with hot water. Place the filter paper and insoluble matter in the original basin, dry in an oven at 100 to 105°C and then ignite until free from carbonaceous matter.

b. If there is a significant amount of insoluble matter left after ignition, digest it for 10 minutes with 10 cm^3 of boiling 50 per cent (w/v) sodium hydroxide solution to remove light siliceous matter. Dilute the solution and separate the residual grit by careful decantation, further dilution and filtration. Wash the grit on to a 9 cm No. 41 Whatman filter paper and continue the washing until free from alkali. Return the paper to the basin and ignite. With a camel hair brush, transfer the grit from the basin to the No. 36 BS sieve and sieve on to the No. 60 BS sieve.

3.4 Reporting

Report the number and nature of the particles retained on each sieve.

4. DETERMINATION OF ORGANIC MATTER SOLUBLE IN ETHER-ALCOHOL MIXTURE

4.1 Outline of method

The nitrocellulose is dissolved in a specially prepared solvent and the solubility determined. In the case of nitrocelluloses with a high nitrogen content (ie a low solubility), the amount of nitrocellulose dissolved in the solvent is estimated. With nitrocelluloses of a low nitrogen content (ie a high solubility), the undissolved residue is determined.

4.2 Low soluble nitrocellulose - minimum nitrogen content 12.75%.

4.3 Apparatus

a. Modified wash bottle fitting consisting of a polythene stopper carrying a normal wash bottle mouthpiece and an all-glass delivery tube and jet. The immersed end of the delivery tube is modified by having the last 2 or 3 cm turned up into a U, (see Figure 1).

b. Alundum crucible (grade RA98) or equivalent. Prepare by igniting in a muffle furnace, soaking in hot aqua regia and washing well with water followed by acetone. Suck the crucible free from solvent and ignite once more.

c. Shaker consisting of a wheel rotating at 15 rev/minute in a water-bath maintained at 15.5 ± 0.5°C, (see NOTE 2).

d. Cylinder, 250 cm^3 stoppered graduated.

4.4 Reagents

a. Diethyl ether. Anaesthetic Ether BP or equivalent quality which is in addition, free from sulphur compounds. Test to confirm the absence of peroxides before use (if appropriate) since their presence constitutes an explosion hazard during the evaporation stage of the solubility determination. The methods used for these two tests are as follows:

i. Sulphur compounds. Acidify 10 cm^3 of the diethyl ether in a glass stoppered cylinder with 1 cm^3 of 10 per cent by volume acetic acid and add approximately 0.2 cm^3 of redistilled mercury. Shake vigorously in the presence of air for one minute and then allow to stand for one hour. The ether-water interface shall remain free from any dark brown or black precipitate since this indicates the presence of sulphur compounds.

ii. Peroxides. Mix 10 cm^3 of 0.005N ferric alum with 50 cm^3 of 0.1N ammonium thiocyanate in a 250 cm^3 glass stoppered conical flask. Titrate with 0.02N titaneous sulphate until the red colour just disappears. Add 10 cm^3 of the ether and shake vigorously for 2 minutes. Peroxides, if present, oxidise the equivalent amount of ferrous thiocyanate and restore the red colour. Stand the mixture for 10 minutes and then titrate with 0.02N titanous sulphate until the red colour disappears. Not more than one drop of titrant should be required.

b. Ethyl alcohol. Alcohol of the quality specified in British Standard 507, 'Ethanol', or equivalent quality, except that the residue on evaporation shall not exceed 0.005 per cent.

Dilute the alcohol with water to 0.830 ± 0.001 sp. gr. at 15.5°C/15.5°C before use.

c. Ether-alcohol solution. Prepare by mixing accurately at 15.5°C two volumes of diethyl ether (a.) with one volume of ethyl alcohol (b.).

4.5 Procedure

a. Transfer 4 ± 0.1 g of the sample to a 250 cm^3 stoppered graduated cylinder which has been calibrated with water. (See NOTE 1).

b. Add 150 cm^3 of the ether-alcohol solution, close the cylinder with a polyethylene stopper and shake vigorously without delay to prevent the nitrocellulose forming a gelatinous mass on the bottom of the cylinder. Continue to shake mechanically for 6 hours. After shaking, stand the cylinder in a water bath or incubator maintained at 15.5 ± 0.5°C (see NOTE 2) and allow the nitrocellulose to settle undisturbed until the supernatent liquid is quite clear, (see NOTE 3).

c. Remove the cylinder from the bath or incubator and read the total volume (V_1) of the contents without delay. Replace the stopper with a second polyethylene stopper carrying the modified wash bottle fitting and adjust the depth of the inlet end of the U so that about 50 cm^3 of the clear supernatant liquid can be removed. By applying gentle pressure from a blowing ball to the mouthpiece of the wash bottle fitting, transfer the 50 cm^3 to a tared platinum dish (weight W_1). Allow the contents of the delivery tube to drain back into the cylinder and read the volume remaining (V_2). Correct the observed volumes for

calibration errors. Stand the dish under a light cover, on the top of a water oven until all the solvent has evaporated, transfer the dish to an oven at 65°C and heat until constant in weight (W_2). Treat the residue with liquid paraffin, ash and carbonate as described in paragraph 2.2b. Carry out a blank determination on an equal volume of the ether-alcohol solution and weigh the carbonated ash. (net weight W_3).

4.6 Calculation and reporting

Calculate the organic matter soluble in ether-alcohol and report as a percentage by weight of sample as follows:

Percentage organic material soluble in ether-alcohol

$$= \frac{(W_2 - W_1 - W_3) \times V_1 \times 25}{(V_1 - V_2)}$$

NOTE 1: When calibrating the cylinder, pay particular attention to the ranges 60 to 90 cm^3 and 150 to 160 cm^3.

NOTE 2: Some National specifications may require the test to be performed at a different temperature, in such cases the purchaser shall nominate the temperature at which the test is to be carried out.

NOTE 3: If the material is disturbed during the settling period (see paragraph b.) the determination must be repeated.

4.7 High soluble nitrocellulose - nitrogen content 10.9 to 12.75 per cent

The organic matter soluble in ether-alcohol may be determined by either Method A or Method B.

4.8 Method A

4.8.1 Apparatus

a. Modified wash bottle fitting consisting of a polyethylene stopper carrying a normal wash bottle mouthpiece and an all-glass delivery tube and jet. The immersed end of the delivery tube is modified by having the last 2 or 3 cm turned up into a U, (see Figure 1).

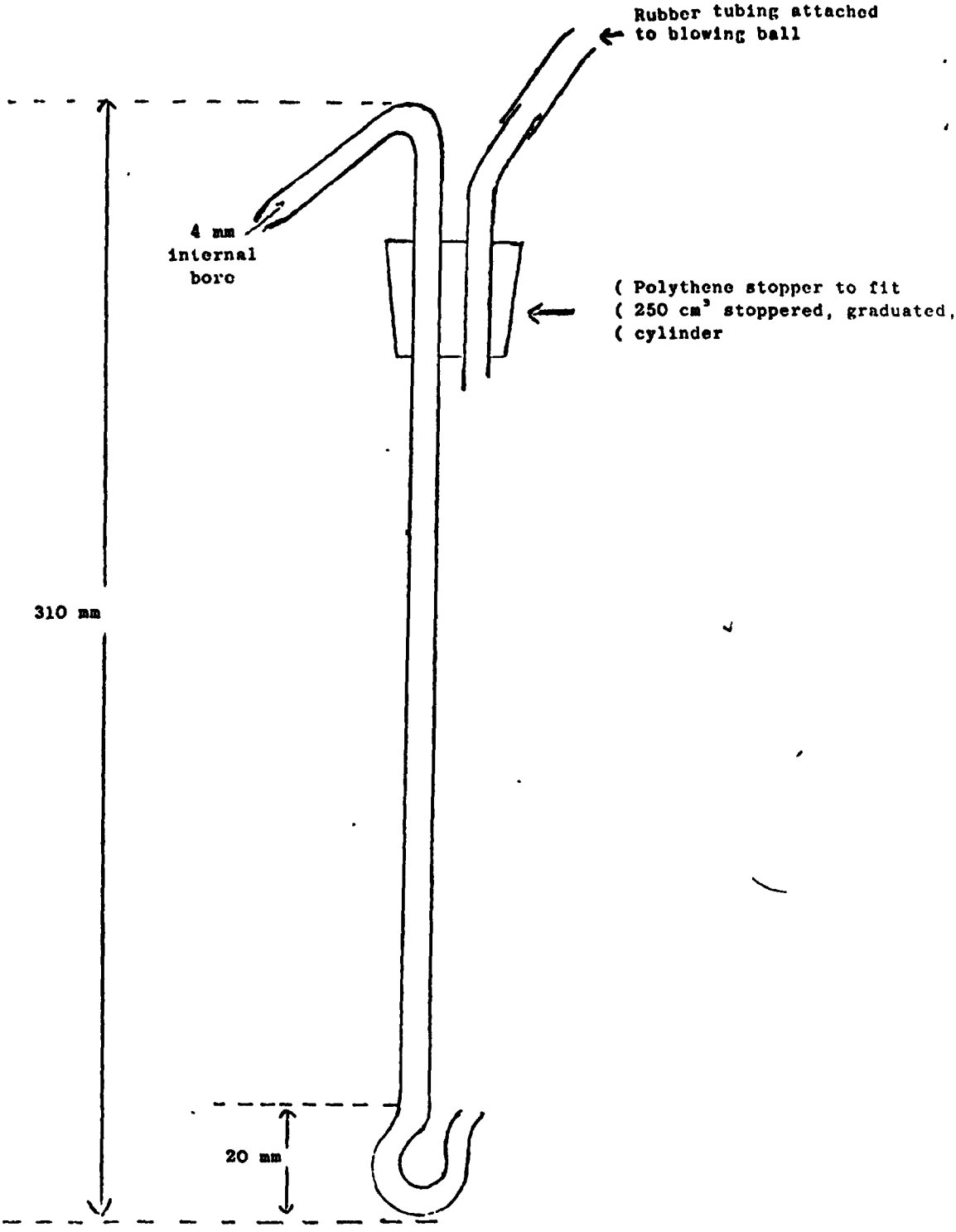
b. Alundum crucible (grade RA98) or equivalent. Prepare by igniting in a muffle furnace, soaking in hot aqua regia and washing well with water followed by acetone. Suck the crucible free from solvent and ignite once more.

c. Shaker consisting of a wheel rotating at 15 rev/minute in a water-bath maintained at 15.5 ± 0.5°C, (see paragraph 4.6, NOTE 2).

d. Cylinder, 250 cm^3 stoppered graduated.

FIGURE 1 (Test 4)

MODIFIED WASH-BOTTLE FITTING FOR ETHER ALCOHOL DETERMINATION



4.8.2 Reagents

a. Diethyl ether. Anaesthetic Ether BP or equivalent quality which is in addition, free from sulphur compounds. Test to confirm the absence of peroxides before use (if appropriate) since their presence constitutes an explosion hazard during the evaporation stage of the solubility determination. The methods used for these two tests are as follows:

i. Sulphur compounds. Acidify 10 cm^3 of the diethyl ether in a glass stoppered cylinder with 1 cm^3 of 10 per cent by volume acetic acid and add approximately 0.2 cm^3 of redistilled mercury. Shake vigorously in the presence of air for one minute and then allow to stand for one hour. The ether-water interface shall remain free from any dark brown or black precipitate since this indicates the presence of sulphur compounds.

ii. Peroxides. Mix 10 cm^3 of 0.005N ferric alum with 50 cm^3 of 0.1N ammonium thiocyanate in a 250 cm^3 glass stoppered conical flask. Titrate with 0.02N titaneous sulphate until the red colour just disappears. Add 10 cm^3 of the ether and shake vigorously for 2 minutes. Peroxides, if present, oxidise the equivalent amount of ferrous thiocyanate and restore the red colour. Stand the mixture for 10 minutes and then titrate with 0.02N titanous sulphate until the red colour disappears. Not more than one drop of titrant should be required.

b. Ethyl alcohol. Alcohol of the quality specified in British Standard 507, 'Ethanol', or equivalent quality, except that the residue on evaporation shall not exceed 0.005 per cent.

Dilute the alcohol with water to 0.830 ± 0.001 sp. gr. at 15.5°C/15.5°C before use.

c. Ether-alcohol solution. Prepare by mixing accurately at 15.5°C two volumes of diethyl ether (a.) with one volume of ethyl alcohol (b.).

4.8.3 Procedure

Transfer 0.5 ± 0.01 g (W_1) of the sample to a 200 cm^3 stoppered graduated cylinder and add 150 cm^3 of the ether-alcohol solution. Close the cylinder with a polyethylene stopper and shake vigorously without delay to prevent the nitrocellulose forming a gelatinous mass on the bottom of the cylinder. Continue to shake mechanically until solution is apparently complete. After shaking maintain the cylinder at 15.5 ± 0.5°C and allow the nitrocellulose to settle (for several days if necessary) until the supernatant liquid is quite clear. Carry out a blank determination at the same time. Remove the polyethylene stopper and replace with the modified wash bottle fitting. By applying gentle pressure from a blowing ball to the mouthpiece of the wash bottle fitting, remove about 100 cm^3 of the clear supernatant liquid without disturbing the sediment. Remove an equal volume of solvent from the blank. Make the remaining solutions in both cylinders up to the original volume with more ether-alcohol solution, shake the cylinders and their contents vigorously once again and leave the solutions at 15.5 ± 0.5°C for the sediment to settle out overnight.

Repeat the removal of nitrocellulose solution, addition of fresh solvent and standing at 15.5 ± 0.5°C once more and finally remove a further 100 cm^3 of supernatant liquid. Treat the blank determination in the same manner. Decant the nitrocellulose solution remaining in the cylinder through a tared alundum crucible (weight W_2) and transfer any sediment left in the cylinder to the crucible by washing with ether-alcohol solution. Wash the residue on the crucible with ether-alcohol solution until the washings leave no residue on evaporation and then dry the crucible at 65°C until constant in weight (W_3). Complete the blank determination in the same manner (blank weight W_4).

NOTE: The residue on evaporation on the ether-alcohol solution is usually negligible and it should only be necessary to carry out the blank determination as a check on each fresh supply of the solvent.

4.8.4 Calculation and Reporting

Calculate the organic matter soluble in ether-alcohol and report as a percentage by weight of the sample as follows:

Percentage organic matter soluble in ether-alcohol

$$100 - \frac{100(W_3 - W_2 - W_4)}{W_1}$$

4.9 Method B

4.9.1 Apparatus

a. Erlenmeyer flask, 300 cm^3, stoppered.

b. Sintered glass crucible, porosity No. 2 or equivalent.

c. Buchner flask, 2000 cm^3.

d. Mechanical shaker assembly for the Erlenmeyer flask.

e. Analytical balance, capacity 100 g, precision 0.1 mg.

f. Desiccator containing $CaCl_2$ desiccant.

4.9.2 Reagents

a. Ethyl alcohol 95° GL or equivalent.

b. Diethyl-ether, pure, free from sulphur compounds. Test to confirm the absence of peroxides before use (if appropriate), since their presence constitutes an explosion hazard during the evaporation stage.

c. Ether - alcohol mixture - add two volumes of diethyl ether to one volume of ethyl alcohol. Adjust the density of the mixture to 758.3 kg/m^3 ± 0.4 by the addition of ethyl alcohol or diethyl ether if necessary.

4.9.3 Procedure

Transfer 1 ± 0.01 g (W_1) of dried nitrocellulose weighed to ± 0.1 mg to the Erlenmeyer flask.

Add 200 cm^3 of ether-alcohol mixture at 20 ± 5°C using a graduated cylinder.

Shake the contents of the flask mechanically for at least 3 hours to dissolve the nitrocellulose.

Filter the contents of the flask through a clean, dry, weighed (W_2) sintered glass crucible attached to a Buchner flask using gentle suction.

Rinse the flask with several portions of ether-alcohol mixture and filter through the sintered glass crucible, transferring any insoluble matter left in the flask to the crucible by means of a jet of ether-alcohol mixture, from a wash bottle.

Wash the crucible thoroughly with the ether-alcohol mixture. Remove any excess ether-alcohol mixture by gentle suction.

Dry the crucible in a well ventilated air circulating oven at 100 ± 5°C for 60 minutes, cool for 30 minutes in the desiccator and weigh (W_3).

4.9.4 Calculation and reporting

Calculate the organic matter soluble in ether-alcohol and report as a percentage by weight of the sample as follows:

$$\text{Percentage organic matter soluble in ether-alcohol} = \frac{100\,(W_1 - (W_3 - W_2))}{W_1}$$

5. DETERMINATION OF ORGANIC MATTER INSOLUBLE IN ACETONE

The organic matter insoluble in acetone may be determined using either Method A or Method B.

5.1 Method A

5.1.1 Apparatus

a. Alundum crucible (grade RA98) or equivalent. Prepare by igniting in a muffle furnace, soaking in hot aqua regia and washing well with water followed by acetone. Suck the crucible free from solvent and ignite once more.

b. Wiley extraction apparatus, see Figure 1.

5.1.2 Procedure

Transfer 1 ± 0.01 g of the sample to 500 cm^3 redistilled acetone contained in a beaker and dissolve by stirring. Allow the solution to stand overnight and filter through the alundum crucible using suction. Wash the beaker well with a jet of acetone from a wash bottle, filtering the washings through the crucible. Transfer the crucible to the extraction apparatus and extract it with acetone for at least 2 days. Remove the crucible, dry it first in air and then in an

oven at 103 to 105°C until constant in weight (W_1). Ignite the crucible in a muffle furnace to destroy the organic matter present, cool and place in a small beaker. Thoroughly wet the interior of the crucible with 1 cm^3 of 5 per cent (w/v) ammonium carbonate solution and heat the beaker and crucible on top of a 100°C oven until the residue is dry. Then heat in an oven at 103 to 105°C as before, until the crucible is constant in weight (W_2). Repeat the carbonation and drying procedure to confirm the completeness of the first carbonation.

5.1.3 Calculation and reporting

Calculate the organic matter insoluble in acetone and report as a percentage by weight of the sample as follows:

Percentage organic matter insoluble in acetone = $(W_1 - W_2) \times 100$.

5.2 Method B

5.2.1 Apparatus

a. Erlenmeyer flask, 300 cm^3, stoppered.

b. Silica sintered crucible, porosity No. 2, or equivalent.

c. Buchner flask, 2000 cm^3.

d. Desiccator containing $CaCl_2$ desiccant.

e. Mechanical shaker assembly for the 300 cm^3 Erlenmeyer flask.

5.2.2 Reagents

a. Acetone, analytical reagent quality or equivalent.

b. Ethyl alcohol, pure, 95° GL or equivalent.

5.2.3 Procedure

Transfer 1.0 ± 0.01 g (W_1) of dried nitrocellulose weighed to ± 0.1 mg to the Erlenmeyer flask.

Wet the nitrocellulose contained in the flask with 10 cm^3 ethyl alcohol and add 100 cm^3 acetone.

Shake the contents of the flask mechanically for approximately 2 hours to dissolve the nitrocellulose.

Filter the contents of the flask through a previously cleaned and ignited Silica crucible attached to a Buchner flask, using gentle suction.

Rinse the flask several times with acetone and filter the liquid through the crucible, transferring any insoluble matter left in the flask to the crucible using a jet of acetone from a wash bottle.

Wash the crucible thoroughly with acetone. Remove any excess acetone using gentle suction.

Dry the crucible in a well ventilated air circulating oven for 90 minutes, cool in the desiccator for 45 minutes and weigh (W_2).

Ignite the crucible in a muffle furnace at 600°C to destroy the organic matter present, cool and place in a small beaker. Thoroughly wet the interior of the crucible with 1 cm^3 of 5 per cent (w/v) ammonium carbonate solution and heat the beaker and crucible on top of a 100°C oven until the residue is dry. Then heat in an oven at 103-105°C until the crucible attains constant weight (W_3).

Repeat the carbonation and drying procedures to confirm the completion of the first carbonation.

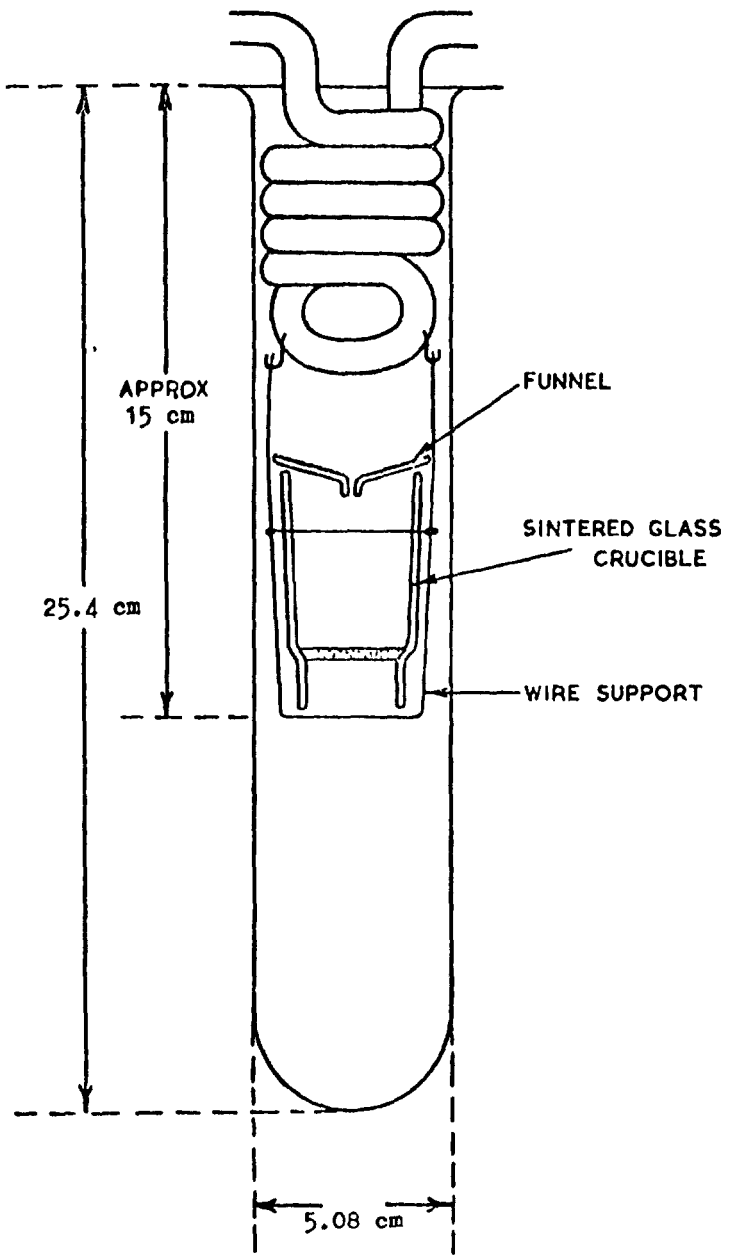
5.2.4 Calculation and reporting

Calculate the organic matter insoluble in acetone and report as a percentage by weight of sample as follows:

$$\text{Percentage organic matter insoluble in acetone} = \frac{W_2 - W_3}{W_1} \times 100.$$

FIGURE 1 (Test 5)

WILEY TYPE EXTRACTION APPARATUS



6. DETERMINATION OF FIBRE SIZE/LENGTH BY SEDIMENTATION

6.1 Apparatus

a. Hard wood rod, 1 inch (2.5 cm) diameter.

b. Cylinders, 250 cm^3 stoppered, graduated.

6.2 Procedure

Transfer 8 g of the sample to a 150 cm^3 beaker, add 30 cm^3 of water and rub it into the nitrocellulose for at least 2 minutes with the hard wood rod. Repeat with 10 cm^3 additions of water (each rubbed in for at least 1 minute) until 50 cm^3 of water have been mixed in and transfer the smooth slurry, obtained after stirring, to the cylinder using a jet of water from a wash bottle to aid the transfer. If any lumps remain in the beaker, break them up by means of the rod and complete the transference of nitrocellulose to the cylinder by washing the beaker with water. Make up the slurry in the cylinder to 200 cm^3 stopper and shake for a few minutes. Place the cylinder on a bench, free from vibration, for at least 30 minutes and then read the volume occupied by the settled nitrocellulose.

6.3 Reporting

Report the volume occupied by the settled nitrocellulose, as the settling test of the sample.

7. VISCOSITY

7.1 Viscosimeter

The viscosimeter employed for this determination shall be the falling ball viscosimeter or approved equal. This viscosimeter consists of a glass tube 14 inches (35.76 cm) in length with an internal diameter of 1 inch (2.54 cm). The lower end of the tube is closed by means of a tightly fitting stopper covered with metal foil. A circle is etched on the tube 2 inches (5.08 cm) from each end, making 2 marks exactly 10 inches (25.4 cm) apart.

7.2 Spheres

Physical properties. The steel balls shall be ordinary 5/16 inch steel ball bearings, having a diameter of 0.793 to 0.795 centimeter and weighing 2.025 to 2.045 g.

7.3 Calibration

To calibrate the spheres, the viscosimeter tube shall be filled with oil or solution of known viscosity (sugar, glycerin, etc) and immersed in a constant temperature bath maintained at 25 ± 0.2°C until its contents reach equilibrium. The balls shall be individually dropped through the tube. The time of passage between the etched circles (see paragraph 7.1) shall be noted. The tube factor K shall be determined as follows:

$$K = \frac{N}{t\,(D - d)}$$

where. N - viscosity in centipoises of solution
t - time in seconds
D - density of the ball g/cm^3 at 25°C
d - density of the liquid g/cm^3 at 25°C.

When one ball has been selected by careful measurement, it shall be weighed and the other spheres chosen shall be of approximately the same weight. The other balls shall be calibrated and only those retained which have the same constant within plus or minus 5 per cent of that of the first ball selected. Specific gravity and density at 25°C; referred to water at 40°C.

7.4 Preparation of specimen

A 20 g portion of the dried nitrocellulose, weighed dried to the nearest 0.01g, shall be transferred to a 16 ounce wide-mouthed bottle, or a 1 litre Kjeldahl flask. A 20 g portion of ethyl alcohol (USP Grade) or equivalent shall be added and the mixture shaken gently to wet the mixture as thoroughly as possible. A 160 g portion of reagent grade acetone (anhydrous) shall be added and the container stoppered. The stopper should be covered with metal foil. The container shall be fastened to a shaking device so that the material will flow rather than mix with air (a rotating wheel with the bottle held at an angle of 45 degrees to the plane of rotation is satisfactory). Mixing shall be continued until solution is complete.

7.5 Procedure

The lower end of the glass viscosimeter tube shall be closed tightly by means of an appropriate stopper covered with metal foil. The tube shall then be filled with nitrocellulose solution until the liquid level is at least 1 inch (2.5 cms) above the upper circle. In order to reach equilibrium, the tube shall then be immersed in a constant temperature bath maintained at 25 ± 0.2°C for at least 1 hour. At the end of this period the tube shall be withdrawn from the bath, wiped dry and then supported in a vertical position by means of a clamp and ring stand. Two or more steel balls, previously calibrated (see paragraph 7.3) shall be allowed to fall separately through the solution, and the time required for each ball to fall from the upper to the lower etched circle shall be noted by means of a stopwatch. The axis of the tube shall be adjusted until the balls fall concentrically, thus showing the glass tube to be in a vertical position. The arithmetic average in seconds, of the time required for the balls to fall from the upper to the lower etched circle shall be reported as the desired value. Calculation for viscosity:

(n) viscosity = KT(D - d)

where. n = viscosity in centipoises
K = constant expressed in centipoises
T = average falling time
D = density of spheres g/cm^3 at 25°C
d = density of liquid g/cm^3 at 25°C.

8. DETERMINATION OF NITROGEN CONTENT

The nitrogen content of nitrocellulose may be determined by either Method A or B.

8.1 Method A - nitrometer method

8.1.1 Outline of method

A prescribed quantity of the sample for test is decomposed by concentrated sulphuric acid in a nitrometer in the presence of mercury. The volume of gas evolved is measured and the nitrogen content of the nitrocellulose calculated.

NOTE. During the determination dangerous mercury and "nitrous" fumes are present and suitable precautions should be taken to keep them below the toxic limits.

8.1.2 Apparatus

a. Room - since nitrogen determinations using the nitrometer involve the measurement of gas volumes, the room in which the estimations are carried out should be free from draughts and rapid fluctuations of temperature. However, because of the toxic danger from mercury and 'nitrous' fumes it is essential that air circulation must be maintained. The floor should be of one continuous surface, free from cracks and with a depression in one corner into which split mercury can be swept and recovered. Preferably, the room shall be thermostatically controlled to 20 ± 2°C and provided with a small extraction fan of the 'Ventaxia' type to change the air without causing draughts.

b. Rack (see Figure 1) - of suitable dimensions, made of stainless or mild steel and incorporating a shelf and a sink, which should be fitted with a mercury trap and running water. Preferably, the rack should be attached to the wall by brackets, thus leaving the floor clear for easier cleaning if mercury is spilled.

c. Nitrometer - 130 cm^3 complying with British Standard 2070, 'Lunge nitrometers' (or equivalent DuPont nitrometer) previously calibrated with mercury and connected by pressure tubing to a mercury reservoir (see Figure 2) whose capacity must be at least 150 cm^3. The tubing metrication should be 5/8 in (1.58 cm) external diameter, having a 3/16 in (0.47 cm) bore and be free from carbonate fillers. It must be cleaned internally to remove any talc coating present on the surface of the bore. Alternatively suitable plastic tubing may be used.

d. Thermometer - calibrated and graduated so that the ambient temperature may be read to 0.1°C. Its bulb must be adjacent to that of the nitrometer and encased in a thin walled glass vessel to protect it from stray draughts.

e. Barometer - calibrated, mercury in glass type, capable of registering the atmospheric pressure to 0.1 mm of mercury.

f. Levelling capstan (see Figure 3) - which may be used to advantage while the internal and external pressures are being equalised.

g. Nitrometer mirrors - may also be used in conjunction with the capstan. The nitrometer mirrors consist of two plane mirrors mounted on a board at a slightly reflex angle so that images of the mercury levels in the reservoir and the measuring tube are superimposed.

8.1.3 Preparation of apparatus

a. Calibration of nitrometer. Fuse a burette-type stopcock of similar glass to the lower end of the nitrometer (see NOTE), thoroughly clean and dry the whole apparatus.

Lightly grease the stopcock and set up the instrument vertically. Fill it with clean, dry mercury and note the ambient temperature. Run mercury from the nitrometer into a tared vessel, until the top of the meniscus is level with the bottom edge of a graduation mark. Weigh the mercury and calculate the corresponding volume at 20°C from tables contained in British Standard 1797, 'Tables for use in the calibration of volumetric glassware' (or equivalent). Compare this volume with the observed volume on the nitrometer and make the appropriate correction.

Repeat the operation for as many graduation marks as necessary.

Remove the stopcock from the lower end of the nitrometer.

NOTE: As an alternative to fusing, the stopcock may be joined to the nitrometer by heavy walled rubber tubing. In this case care must be taken to see that the tubing is put on firmly and the glass of the stopcock and nitrometer are in contact. The joint should be clamped to avoid strain from the weight of mercury above it. If hot air is used for drying, make sure that the apparatus has settled down to room temperature before beginning standardisation.

b. Assembly of nitrometer. Connect the clean, calibrated nitrometer to the reservoir by 5-6 ft (approximately 2 metres) of the pressure tubing and wire the two ends firmly to the glassware. Tape over the wire to protect the operator's hands during the subsequent determinations and mount the nitrometer and reservoir on the rack by means of split retort rings covered with rubber tubing (see Figure 3). Lightly grease the key of the nitrometer stopcock (see NOTE 1), replace it in the barrel and open the stopcock to the cup.

Pour mercury into the reservoir until there is enough present to fill the nitrometer and tubing and leave a reasonable excess in the reservoir. Slowly raise the reservoir from its ring and thus fill the nitrometer with mercury. Shut the stopcock and lower the reservoir back to its original position.

Nearly fill the nitrometer cup with concentrated sulphuric acid, open the stopcock and allow the acid to run into the nitrometer. Thoroughly rinse the mercury with the acid by shaking the nitrometer (see NOTE 2), replace the nitrometer on the rack and allow the acid to drain upwards through the mercury.

Do not allow the acid to come in contact with the tubing. Open the stopcock to the capillary outlet and discharge the acid into a beaker by raising the reservoir. Lower the reservoir to its original position and then repeat the rinsing once more. Drain and discharge the acid as before, but this time fill the outlet and both bores of the stopcock with mercury before finally closing the stopcock. Lower the reservoir 20-30 cm below the cup to check that the stopcock is gas tight, (see NOTE 3). Raise the reservoir to its original position and wipe the excess acid quickly from the inside of the cup by using filter paper. Cover the outlet tube with a rubber cap and the nitrometer is ready for use.

NOTE 1: The grease should not evolve gas when in contact with concentrated sulphuric acid and should be applied sparingly to avoid fouling the nitrometer.

NOTE 2: The shaking is done as follows.

Remove the nitrometer from the stand, hold the bottom of the tube firmly in one hand and keep it stationary. Cup the bulb in the other hand, with the key of the stopcock held in place by the heel of the thumb and shake the nitrometer smartly through an arc from shoulder to waist level.

NOTE 3: If air leaks are observed, the stopcock must be cleaned, re-greased and all the capillaries filled again with mercury.

8.1.4 Procedure

a. Weigh approximately, a suitable amount of the sample (see NOTE 1) into a tall, stoppered weighing bottle (approximate dimensions 4 x 2 cm) and dry for one hour in an oven at 100°C. Cool in a desiccator and weigh the bottle and contents accurately (W_1).

b. Pour 15 cm^3 of the 94 per cent sulphuric acid into a 25 cm^3 measuring cylinder and transfer a few drops of the acid to the nitrometer cup so that the constricted portion between the cup and the stopcock is filled. Remove the stopper from the weighing bottle and transfer the sample to the cup by inverting

the bottle into it, taking care that no fine dust is lost in the operation. Replace the stopper carefully and reweigh the bottle (W_2) to give, by difference, the weight of sample taken.

c. Add approximately 5 cm^3 of the acid in the cylinder to the nitrometer cup, quickly stir the mixture with a glass rod and draw the resulting suspension into the nitrometer as quickly as possible. Wash the rod and the cup free from nitrocellulose by using successive small portions (2 cm^3) of the remaining acid and drawing each portion separately into the nitrometer. Allow the nitrometer to stand for 15 minutes and in the meantime close the cup with a rubber stopper and check that the rubber cap is in position on the outlet tube. Remove the nitrometer from its stand and depending on the nitrocellulose under test, shake as follows:

i. Nitrocellulose of nitrogen content not less than 13.35 per cent N

Shake for 1 minute, stand for 2 minutes, shake for 2 minutes, stand for 2 minutes and finally shake for 2 minutes.

ii. Nitrocellulose of nitrogen content less than 13.35 per cent N

Shake for 1 minute, stand for 2 minutes and finally shake for 2 minutes.

d. Return the nitrometer to the stand, remove the rubber stopper and adjust the pressure of the gas in the nitrometer (see NOTE 2) to approximately atmospheric by lowering the reservoir until the level of the mercury which it contains is higher than that in the nitrometer, by one seventh of the difference between the acid and mercury levels in the nitrometer. Leave the nitrometer to stand for 20 minutes and then adjust the position of ther reservoir accurately so that there is no movement of acid in the bore of the key when the stopcock is opened.

Close the stopcock and allow any acid which has entered the nitrometer during 'levelling', to drain for 5 minutes. Re-open the stopcock carefully and re-adjust the pressure if necessary. Note the volume (V) of the gas (reading the bottom of the acid meniscus), the ambient temperature (T) and the barometric pressure (P).

NOTE 1. Different nitrocellulose vary considerably in nitrogen content and therefore varying weights of sample must be used in order to obtain reasonable gas volumes for measurement. The following table shows the weights of nitrocellulose required for different nitrogen contents.

Approximate nitrogen content (percentage N)	Weight taken (g)
10.9 - 11.4	0.62 - 0.63
12.1 - 12.3	0.55 - 0.56
12.5 - 12.8	0.53 - 0.54
13.0 - 13.4	0.51 - 0.52

NOTE 2: The pressures can be adjusted very accurately, by means of the levelling device shown in Figure 3. The operator's hands should be kept as far as possible from the nitrometer bulk during levelling, to prevent heat transfer to the gas and as little acid as possible should be left in the bore of the key when levelling is completed.

8.1.5 Cleaning of nitrometer

After each determination is completed, clean out the nitrometer as follows:

Remove the rubber cap from the outlet tube, open the stopcock to the outlet tube and expel the gases and the acid sludge in the nitrometer into a beaker, by raising the reservoir. Close the stopcock, lower the reservoir to its original position and nearly fill the nitrometer cup with concentrated sulphuric acid. Open the stopcock, run the acid into the nitrometer by suitable manipulation of the mercury levels and close the stopcock once again. Remove the nitrometer from the stand and dislodge the sludge from the walls of the nitrometer by gentle shaking. Replace the nitrometer, allow the acid to drain upwards through the mercury and then expel it as before. Repeat the cleaning with acid and leave the second rinsing to drain in the nitrometer until the apparatus is required for the next determination.

8.1.6 Calculation and reporting

a. Correct the observed volume of gas in cm^3 (V), for nitrometer scale error = V_1.

b. Correct the observed barometric pressure in mm of mercury (P), to 0°C and for latitude = P_1.

c. Correct the observed termperature in °C (T), for thermometer scale error = T_1.

d. Obtain the percentage ash content (A).

e. Calculate the nitrogen content and report as a percentage by weight of the dry, ash free sample as follows:

$$\text{Percentage nitrogen content} = \frac{V_1 \times P_1 \times 2.247}{(273 + T_1) \times (W_1 - W_2) \times (100 - A)}$$

8.2 Method B - Devarda's alloy method

8.2.1 Outline of method

The sample which has been previously dried is treated with potassium hydroxide in the presence of hydrogen peroxide (110 vol). The potassium nitrate formed by the saponification of the nitro esters contained in the sample is then reduced by Devarda's alloy with the formation of ammonia. The ammonia is distilled into an excess of N/3 sulphuric acid and determined by back titration with N/6 sodium hydroxide solution.

8.2.2 Apparatus

a. The saponification, reduction and distillation is carried out in the same flask. A 500 cm^3 flat bottomed flask made of borosilicate glass is used and to which is fitted a Wagner tube (see Figure 4). The dimensions and shape of the tube are important and should conform as near as possible with those shown in Figure 4.

b. Receiving flask consisting of a 500 cm^3 flat bottomed conical flask.

c. Burette, Grade A, 10 cm^3, graduated in 0.05 cm^3.

d. Pipette, Grade A, 25 cm^3.

e. Balance, capacity 100 g, accuracy 0.1 mg.

f. Boiling water oven.

g. Hot plate set at 50-80°C.

8.2.3 Reagents

a. Potassium hydroxide solution - dissolve 750 g potassium hydroxide in 1 litre of water.

b. Hydrogen peroxide (110 volumes).

c. Devarda's alloy - material with a specific surface area of 1800-3200 cm^2/g has been found to be the most suitable.

d. N/3 sulphuric acid.

e. N/6 sodium hydroxide solution.

f. Indicator solution (Tacchiro's Reagent) - 0.1% alcohol solution of a mixture of 8 g methyl red and 3 g methylene blue.

g. Alcohol 95° GL.

8.2.4 Procedure

a. Weigh approximately, a suitable amount of the sample (see NOTE 1) into a tall stoppered weighing bottle (approximate dimensions 4 x 2 cms) and dry for 1 hour in an oven at 105°C. Cool in a desiccator and weigh the bottle and contents accurately (W_1). Remove the stopper from the weighing bottle and transfer the sample to the saponification flask taking care that no fine dust is lost in the process. Replace the stopper carefully and re-weigh the bottle (W_2) to give the weight of the sample by difference.

b. Add to the saponification flask 10 cm^3 of alcohol, 10 cm^3 of hydrogen peroxide and 20 cm^3 of potassium hydroxide solution by means of a burette or a measuring cylinder ensuring that the liquids flow down the wall of the flask and at the same time rotating the flask. Place the flask on the hotplate taking care that no particles of nitrocellulose adhere to the walls of the vessel and heat for 10-20 minutes. Add 130 cm^3 distilled water and heat the contents of

the flask over a bunsen burner until the hydrogen peroxide is destroyed, this is indicated by absence of large bubbles of oxygen being given off.

c. Pipette 25 cm^3 of N/3 sulphuric acid into the receiving flask.

d. Add 5 g Devarda's alloy to the saponification flask and assemble the apparatus as shown in Figure 4 with the minimum of delay. Cool the saponification flask in a water bath containing ice to reduce the rate of reaction, allow the reaction to continue in the cold for approximately 30 minutes during which time the reduction should be complete. Remove the cooling bath and heat the saponification flask, slowly at first, until approximately 2/3 of the liquid has been distilled. Disconnect the Wagner tube from the saponification flask and wash down with distilled water into the receiving flask, remove the tube from the flask.

e. Titrate the solution in the receiving flask with N/6 sodium hydroxide solution using 4 drops of Tacchiro reagent as indicator. Record the volume of N/6 sodium hydroxide using (t ml).

f. Carry out a blank test on the reagents used by the above procedure and record the volume of N/6 sodium hydroxide used (T ml).

8.2.5 Calculation and reporting

Calculate the nitrogen content and report as a percentage by weight of the sample as follows:

$$\% \text{ Nitrogen} = \frac{0.2335 \times (T - t)}{(W_2 - W_1)}$$

NOTE 1. The weight of sample required to produce sufficient ammonia to nearly saturate the 25 cm^3 of N/3 sulphuric acid is dependent on the nitrogen content of the sample - see below:

% Nitrogen content	Weight sample required - g
10.9 - 11.4	0.96 - 1.00
12.1 - 12.3	0.88 - 0.92
12.45 - 12.75	0.85 - 0.90
13.0 - 13.25	0.83 - 0.87
13.35 minimum	0.80 - 0.85

FIGURE 1 (Test 8)

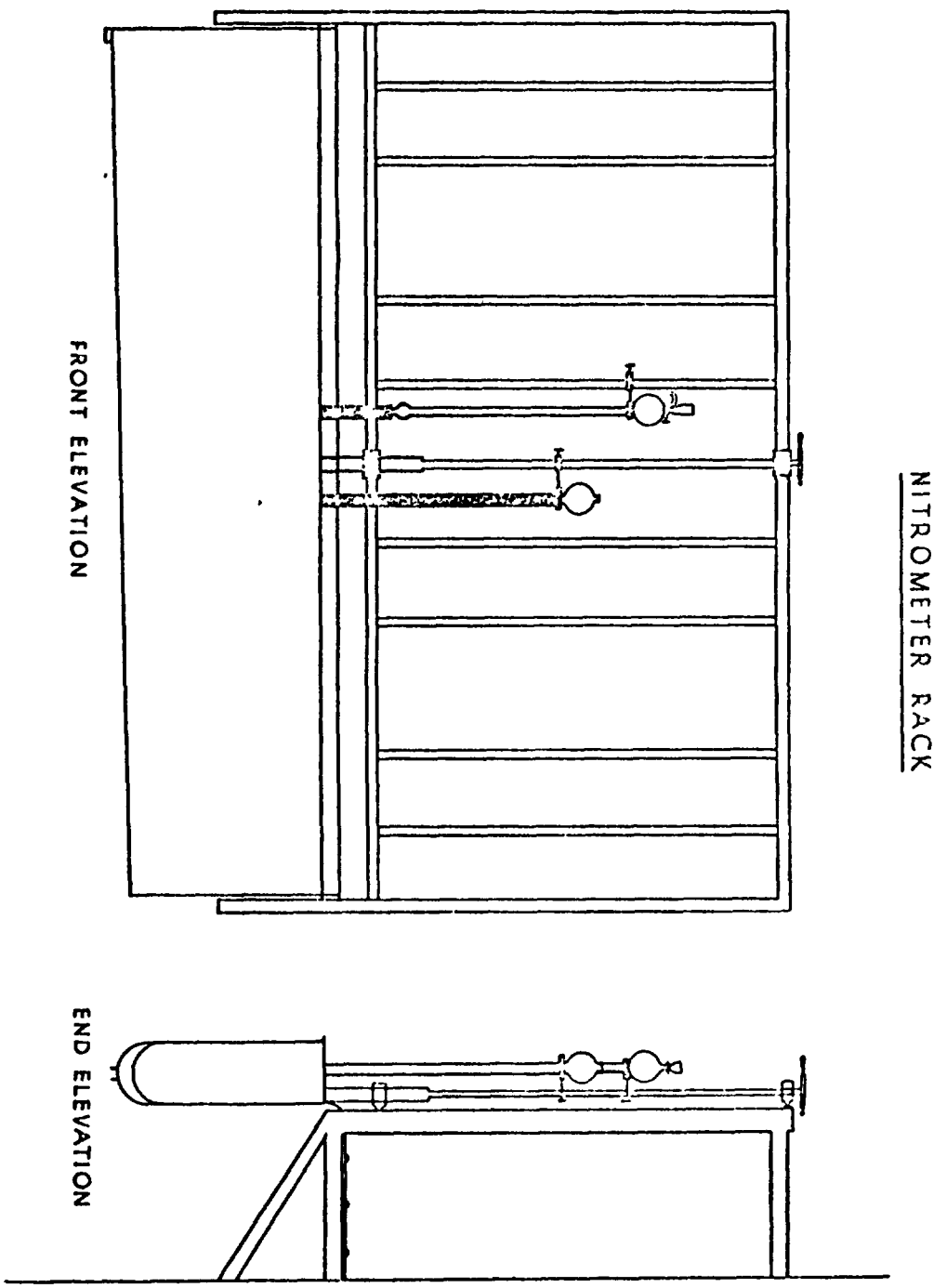
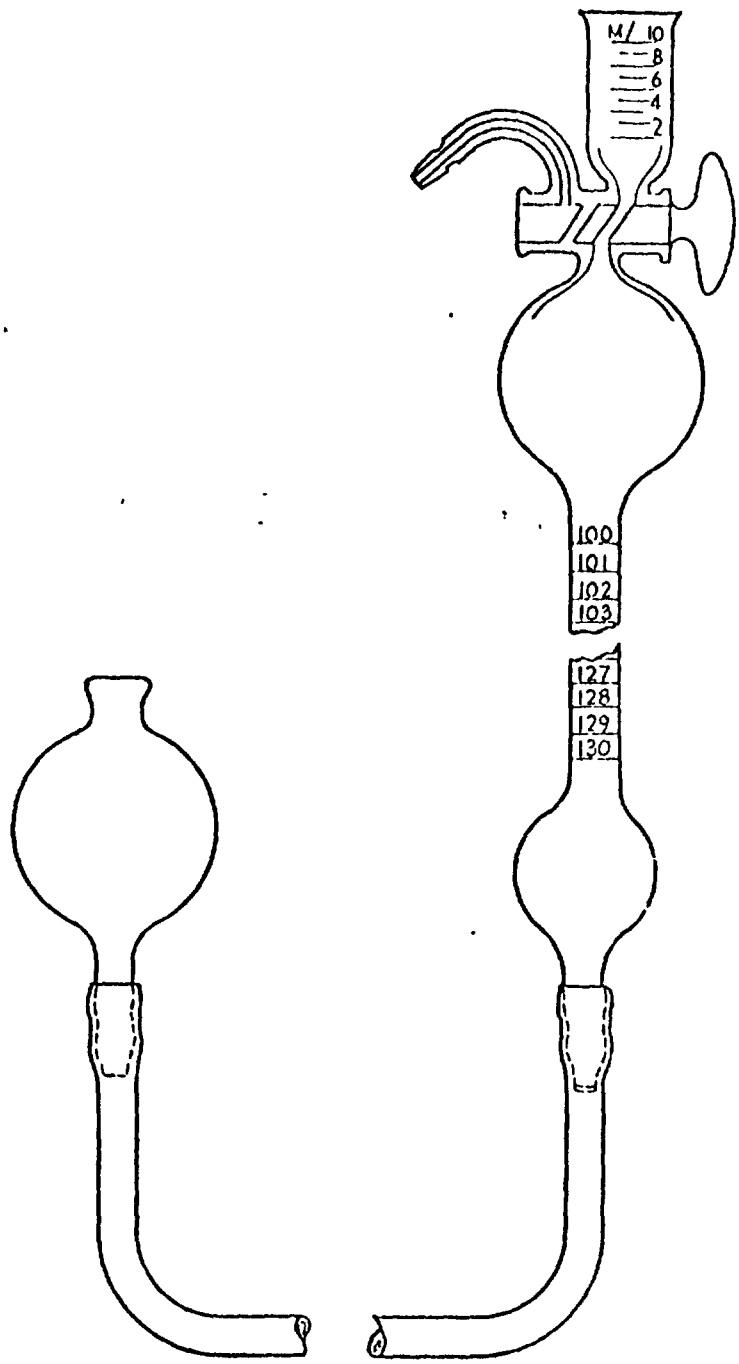


FIGURE 2 (Test 8)

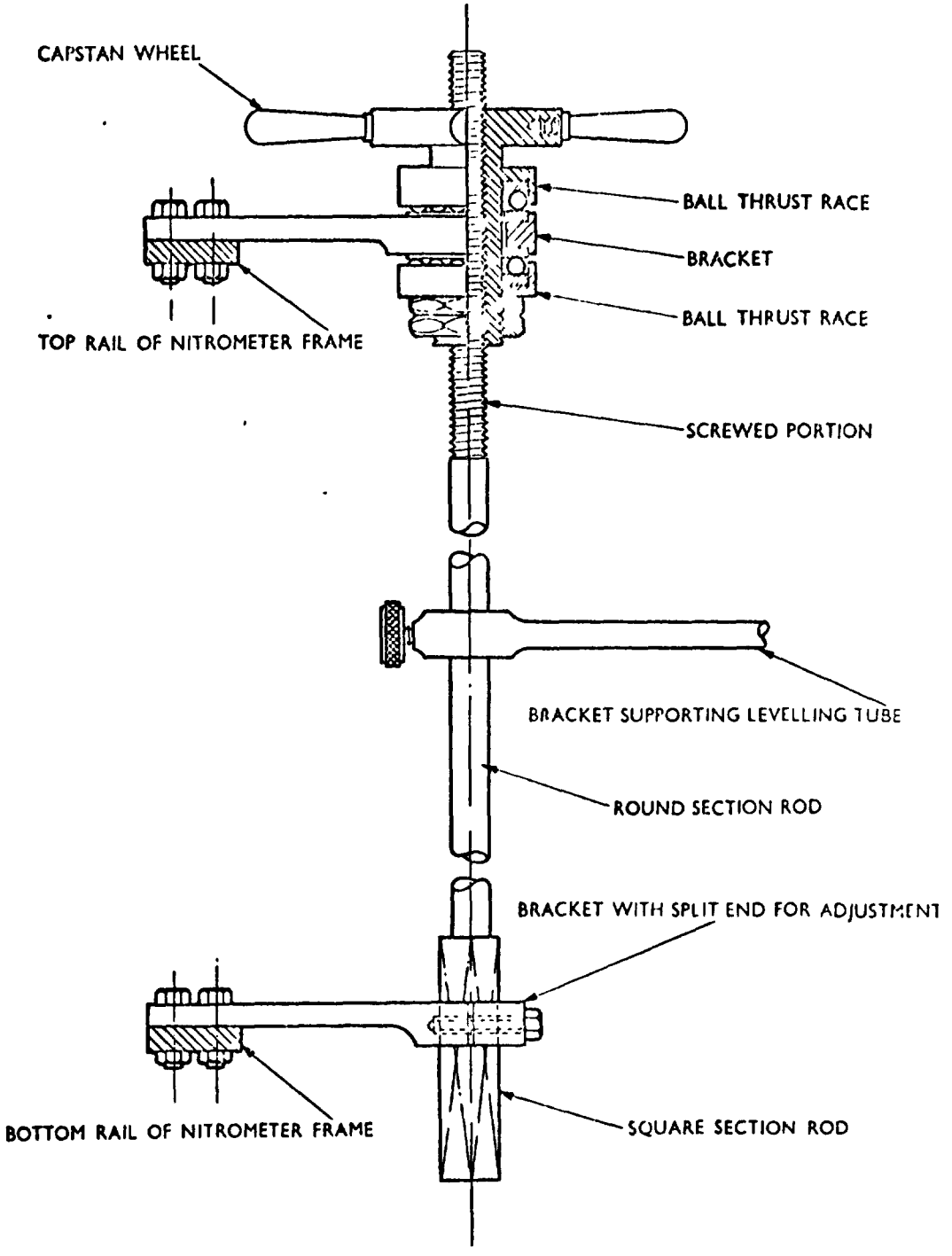
LUNGE NITROMETER
130 ml Size



ANNEX A to
STANAG 4178

FIGURE 3(Test 8)

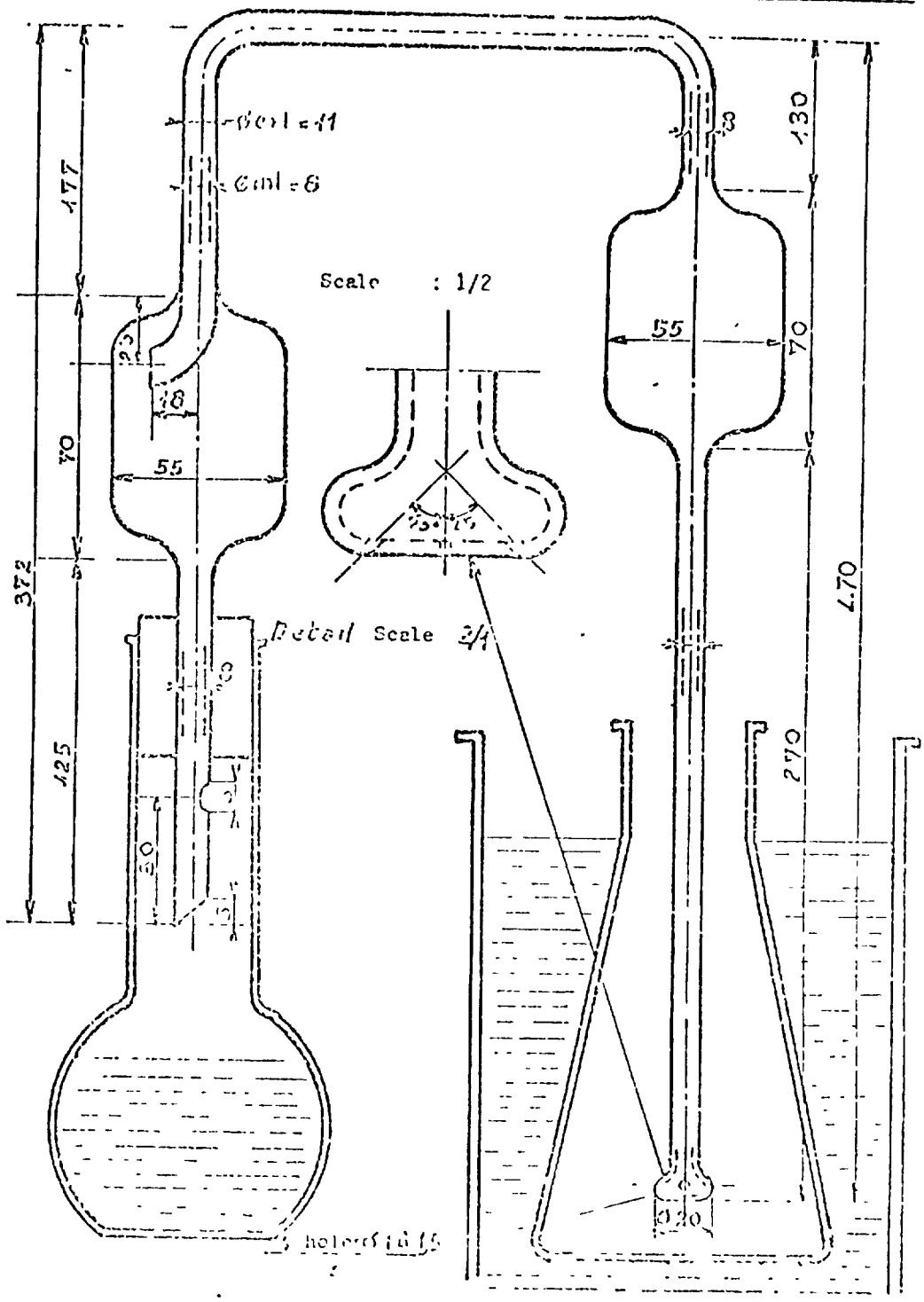
NITROMETER CAPSTAN



ANNEX A to
STANAG 4178

FIGURE 4 (Test 8)

APPARATUS FOR THE DETERMINATION OF NITROGEN BY THE DEVARDA'S ALLOY METHOD



Dimensions in millimetres

9. DETERMINATION OF STABILITY BY THE 132° STABILITY TEST
(modified Bergmann and Junk stability test)

9.1 Outline of method

The 132°C stability test is used to measure the stability of nitrocellulose. A prescribed quantity of the material to be tested is heated in a specially designed glass apparatus for 2 hours at 132°C. The extent of decomposition is measured by absorption of the evolved oxides of nitrogen in water and their determination in the solution so obtained.

9.2 Apparatus

a. Stability bath - a heating bath capable of maintaining the temperature of the stability tubes at 132 ± 0.2°C.

b. Thermometers - special stability test thermometers are used for recording the temperature of the bath. The thermometers are large bulb, short range instruments graduated in 0.1°C between their limits of 125-140°C.

c. Stability tubes - details of which are shown in Figure 1 shall be made of colourless resistance glass. Each new supply must be examined as to the quality of glass and be seasoned before use.

d. Gauge - calibrated at 6 in (15 cm) for measuring the depth of the stability tube.

9.3 Reagents

a. General - unless there is an instruction to the contrary, the use of chemicals of Analytical Reagent quality is to be understood. If water is used as a reagent, it shall be freshly distilled or freshly boiled and cooled distilled water or deionised water of equivalent purity.

b. Non-reactive grease - prepare by dissolving 35 g of paraffin wax in 65 g of warm liquid paraffin and allowing the resulting mixture to cool.

9.4 Procedure

a. Preparing the stability tubes - examine approximately 10 per cent of each new supply by rinsing the tubes with several changes of warm water and adding to each, 20 cm^3 of water followed by 50 cm^3 of 0.1 per cent w/v narcotine hydrochloride solution. Close each tube by a bulb to prevent access of dust and place them together in a tall beaker filled to the same level with hot water. Place the beaker on a hot plate in an atmosphere free from acid and other chemical fumes and boil gently for 1½ hours, maintaining the water level in the beaker. Remove the tubes and after wiping the outside, examine them in a good light for the presence of lustrous needles of narcotine base in the solution. The quantity of free narcotine deposited in the form of fine lustrous needles is a measure of the amount of alkalinity liberated from the surface of the glass and not more than the faintest trace of free narcotine should be visible if the tubes are of satisfactory quality. After this examination, season each new tube before use by rinsing first with water and then filling it to the neck with cold 0.01N hydrochloric acid. Alow it to stand for 12 hours, rinse it thoroughly with water and dry.

b. Calibrating the thermometers - calibrate the thermometers used in the tests, against a suitable standard thermometer. Record any correction and the date on which it was obtained on a tab affixed to the thermometer. Repeat the calibration at intervals of three months.

c. Performing the stability test - transfer three portions of 2 ± 0.01 g each of the sample, to stability tubes. Brush down any material adhering to the sides of the tubes and if the nitrocellulose occupies a greater length than 5 cm, compress it to that length by means of a flatheaded boxwood rod. Set one of the tubes aside as a blank, lubricate the joints of the other two tubes with the non-reactive grease and fit these with absorption cups. Seal off the cups with 8-10 cm^3 of water taking care to keep the contents of the stability tubes dry. By means of a suitable cork with a central hole, support each tube vertically in one of the holes of the bath, so that it is immersed to a depth of 6 in (15 cm) below the metal top of the bath. Maintain the tubes at a temperature of 132 ± 0.2°C for 2 hours unless pronounced fuming is observed, then the test should be stopped immediately and the duration of the heating period noted. Confirm the maintenance of a steady bath temperature by leaving two of the thermometers in position on opposite sides of the bath during the test and keep the temperature of the air above the bath in the vicinity of the absorption cups, below 40°C during this period. Remove the stability tubes from the bath, add a further quantity of water to the cups and allow the apparatus to cool to room temperature behind a safety screen. Water will be drawn into the tubes during this time. Remove the cups from the tubes and transfer the water from each cup, quantitatively to a 500 cm^3 conical flask. Add 20 cm^3 of 0.1N hydrochloric acid to each of the stability tubes and to the blank. Stopper the tubes and shake for 15 minutes. Plug the tops of the stems of three 6 cm filterfunnels with pads of absorbent cotton wool, flush the pads with water and filter the contents of the tubes through them into the appropriate flask. Wash the nitrocellulose thoroughly on the filter funnel and add 25 cm^3 of 0.1N sodium hydroxide to each of the flasks. Adjust the volumes in the flasks to be roughly equal and titrate with 0.1N hydrochloric acid using 3 drops of methyl orange as indicator. Match the colours of the test solutions at the end point with that of the blank. (Net titre = T).

9.5 Calculation and reporting

Calculate the stability and report as milligrammes of nitrogen evolved per gramme of sample, as follows.

Milligrammes nitrogen per gramme nitrocellulose = 0.7T × factor of 0.1N HCl.

NOTE: Alternatively the stability may be reported as the number of cm^3 of 0.1 NaOH consumed per gram of nitrocellulose.

10. DETERMINATION OF STABILITY BY THE HEAT TEST

10.1 Outline of method

A prescribed quantity of the sample for test is heated in a special tube immersed in a water bath maintained at the temperature prescribed in the purchasing Specification. A starch potassium iodide paper is suspended in the tube and the length of time, in minutes, which elapses before the tint of the line produced on the paper equals that of a standard is recorded as the heat test of the nitrocellulose.

10.2 Apparatus

a. Water bath - a water bath of suitable design capable of being maintained within ± 0.3°C of the temperature prescribed in the purchasing Specification. A recommended bath is described in 10.8.

b. Thermometer - thermometer F 100 C/100 complying with the requirements of BS 593, or equivalent.

c. Dropping bottle - a 60 cm^3 kali type bottle with a perforated rubber stopper carrying the glass rod complying with the requirements of specification DEF-4515, 'Rod, glass, dropping Mark I (Apparatus, Abel Heat Test)'.

d. Gauge - the heat test paper gauge depicted in Figure 4.

e. Piercing plate - the perforated glass plate depicted in Figure 2.

f. Rings - a supply of the rubber rings depicted in Figure 3. The rubber shall be of good quality and compatible with the explosive under test.

g. Hook assemblies - a supply of rods and hooks complying with the requirements of specification DEF-4512. 'Rod, glass, platinum hook, Mark II (Apparatus, Abel Heat Test)', mounted in the rubber stoppers depicted in Figure 4. The rubber shall be of good quality and compatible with the explosive under test.

h. Test tubes - a supply of test tubes complying with the requirements of specification DEF-4514, 'Test tube, Mark II (Apparatus, Abel Heat Test)'.

j. Funnel - the aluminium powder-funnel depicted in Figure 4.

k. Standard tint paper - a standard tint paper manufactured and supplied by Materials Quality Assurance Directorate, Royal Ordnance Factory, Bishopton (or equivalent) and issued not more than one year before the date of use. The paper is contained in a colourless glass tube, stoppered and enclosed in a cylindrical cardboard carton marked with the date of issue.

m. Cotton wool - absorbent cotton wool BPC is suitable.

n. Lighting - an artificial light source of the colour matching type giving an illumination between 430 and 1600 lux, placed so that the test line is read by reflected light. Typical arrangements, giving an illumination of 1100 lux approximately, are described in 10.8.

Alternatively, the test line may be viewed in reflected north daylight (south daylight in the southern hemisphere) of adequate intensity.

10.3 Reagents

a. Heat test papers - papers manufactured and supplied by Materials Quality Assurance Directorate, Royal Ordnance Factory, Bishopton (or equivalent) and issued not more than one year before the date of use. The papers are supplied in amber-coloured tubes, each containing 100 papers and marked with a batch number and date of issue.

b. Glycerine-water mixture - a 50% (v/v) mixture of glycerine and water.

10.4 Precautions - Special

a. Heat test papers - when not in use keep the tube of papers tightly stoppered, cool, dry, and in the dark. Remove the papers from the tube singly, using forceps. Discard the selected paper if, subsequent to removal, it is not immediately prepared and inserted in the heat-test tube as described in clause 10.6. Avoid undue exposure to light and do not touch the papers with the fingers. Since the papers are very sensitive and readily affected by agents other than the oxides of nitrogen they are primarily intended to detect, keep the atmosphere free from reactive vapours. Discard any tube of papers not used within one year of the date of issue.

b. Standard tint paper - renew the standard tint paper annually and discard any paper which has a date of issue more than one year old. Do not remove the standard tint paper or the stopper from the tube at any time. When not in use, keep the standard tint paper in its carton or otherwise protect it from light.

c. Heat test laboratory - keep the laboratory free from chemicals likely to cause reactive fumes. Ensure that the background of the bath is free from any yellow tint; it is advisable to have a pale neutral-grey matt finish on the whole surface of the walls and ceiling.

d. Lighting - the traditional standard lighting for the observation of the heat test end point is north daylight of adequate intensity, and the test paper is viewed by reflected light. The limits of this natural standard cannot be defined with any accuracy, and the artificial daylight standard described in paragraph 10.8 is recommended.

e. Water or solvent vapour - the heat test is an empirical method based on a time of reaction which can be greatly affected by the conditions of test and the state of the sample. Moisture or solvents, commonly present in explosives, can lengthen or shorten the time of test, depending of their concentrations and other factors. It has been established, for example, that to obtain the minimum heat test time of nitrocellulose, the test sample must have a moisture content between 1% and 2%. A low moisture content inhibits the formation of the acids necessary to complete the reaction, and leads to longer heat test times on the other hand too much water dilutes the acidic gases, with similar results. In the latter case, moisture may condense on the inside of the test tube and hinder the viewing of the end-point.

f. Carry out the heat test without delay after the completion of the prescribed sample preparation.

10.5 Sample preparation

If the nitrocellulose is received dry, the heat test is carried out in duplicate without preliminary preparation, but usually the material is received in the alcohol or water-wet state and requires drying so that it contains 1% to 2% moisture. Normally this may be done in the following manner:

Lightly rub approximately 40 g of the wet sample with a bone, horn, or wood spatula, to break down large aggregates. Spread the rubbed sample in a uniform thin layer on an aluminium tray lined with filter paper, and air-dry the material overnight at a relative humidity below 50% and at an ambient temperature of 23 ± 3°C.

If the sample is very wet, a preliminary pressing may be necessary. Carry this out in the following manner:

Place the material (approximately 50 g) between layers of thick filter paper which must be free from chemical impurities, and put it under a hand screw press. Press firmly for 3 minutes, and if necessary repeat the pressing after renewing the filter paper. Tease out the nitrocellulose loosely with the fingers, rub it down with the spatula, and dry it by the procedure previously described.

10.6 Procedure

Weigh two 1.3 g portions of the dry nitrocellulose into a scoop, and transfer each portion to a separate heat-test tube with the aid of the powder funnel. Tap each tube gently in order to settle the material, and remove as much nitrocellulose as possible from the sides of the tubes. If necessary, compress the sample in the tubes to a depth of 3 cm by means of a glass rod with a flat end, and clean the remaining nitrocellulose off the sides of the tube with a piece of muslin. Carry out the heat test in duplicate.

Take each heat-test tube containing the sample, fit it with a rubber ring, and adjust the latter so that its lower edge coincides with the bottom engraved line on the test tube. Using forceps, remove a test paper from its amber-coloured glass tube and position it, smooth side uppermost, on the perforated glass plate ready for piercing. Pierce the paper with a suitable needle at the point halfway across its width and near one end. Hold the paper in the forceps by the unperforated end, in an inclined position with the ends horizontal and the perforated end uppermost. Withdraw a suitable amount of the glycerine-water mixture on the glass rod of the dropping bottle by dipping the rod in the mixture to a depth of approximately 3 cm. Hold the rod parallel with the upper edge of the paper, and tilt them both slightly from the horizontal so that any excess liquid flows away from the tip of the rod. Apply the side of the rod, a little way from the end to the upper edge of the test paper, and draw it rapidly and evenly down the surface of the paper to a distance of approximately 0.4 cm from the upper edge. The distance depends to some extent upon the amount of liquid on the rod, and the liquid is to be applied to the paper in a single smoothly performed operation. Maintain the paper in its oblique position until the excess liquid on the surface has been absorbed. The amount of liquid applied, which experience helps in judging, shall be such that the dividing line between the wet and dry portions of the paper at the completion of the test is 0.9-1.2 cm from the upper edge and parallel to it.

Suspend the paper on the platinum hook of the assembly, and insert the rubber stopper in the heat-test tube so that the paper hangs vertically. Adjust the stopper so that its bottom edge is level with the top engraved line of the tube, and allow the tube to stand for about 1.5 minutes, by which time it will be possible to judge whether the paper has been wetted satisfactorily. Discard the paper if it is clearly under or over wetted, and replace it with a fresh one. Adjust the rod carrying the satisfactorily wetted paper until the wet-dry boundary is opposite the middle engraved line on the test tube. Check that the bath is at the correct temperature for the type of explosive being tested, and insert the tube in the bath within 3 minutes of wetting the test paper. Note the time of insertion on a stop clock, and maintain the temperature of the bath within ± 0.3°C of the nominal test temperature until the brown line appearing at the wet/dry boundary on the test paper equals in colour the brown line on the standard tint paper. The coloured lines produced in the test vary slightly

in width, regularity, and definition, and occasionally in the uniformity of the colour. All these factors combine to influence the appearance of the line, but special care must be taken to disregard their effect and to consider only the colour of the lines. The test is taken as being complete when the shade and intensity of a small area in the darkest part of the coloured band are considered to be equal to those of the standard tint. A piece of white filter paper held behind the test tube enables the end point to be judged more precisely. Note the time elapsed between insertion and completion, and record this as the Heat Test of the sample, together with the temperature and date of test. Remove the tube from the bath and the paper from the assembly, and examine the paper with the gauge to confirm that the test line is within the specified limits. If the line is outside the gauge limits, repeat the test.

10.7 Cleaning and maintaining the apparatus

a. Clean all tubes and bottles used for heat test work in the following manner before use, and prior to re-use. Wash out thoroughly with hot water (and detergent if necessary), using a test-tube brush. Rinse thoroughly and allow to drain for a few minutes. Repeat the rinsing with distilled water and finally rinse with acetone. Dry in an oven at approximately 100°C, and allow to cool at room temperature before using again.

b. Clean the rubber stoppers periodically by scouring them in hot water with pumice stone, rinsing with hot distilled water, and drying in an oven at approximately 50°C.

c. Keep the glass rods carrying the hooks lubricated with glycerine-water mixture so that there is a sliding fit between rod and stopper.

10.8 Arrangement of bath and lighting for heat testing

10.8.1 Bath

a. A suitable electrically-heated bath of the traditional Abel pattern, depending on convection for maintaining uniformity of temperature, is described in Admiralty Specification B77B and drawings NOD 7071 and NOD 7071/1.

b. A recommended alternative design for bath consists essentially of a lagged rectangular tank about 270 x 360 x 240 mm deep, incorporating a levelling device which keeps the water level 6 mm below the underside of the lid. Temperature control is maintained by a commercially available stirrer-heater unit. The lid is drilled to take up to 12 heat-test tubes, and is fitted with a device for holding the tubes in a vertical position.

10.8.2 Lighting

Consistent artificial lighting is preferred to 'north' daylight which may vary considerably in quality and intensity. Two forms of artificial lighting systems are recommended.

a. A fluorescent tube, of colour matching type, attached horizontally to the front of the bath by adjustable brackets so that the tube is about 120 mm above the surface of the bath, as described in MQAD Technical Paper 419. The test papers can then be viewed by reflected light through the gap between the bottom

edge of the reflector and the top of the bath. Details of the lighting assembly are as follows:

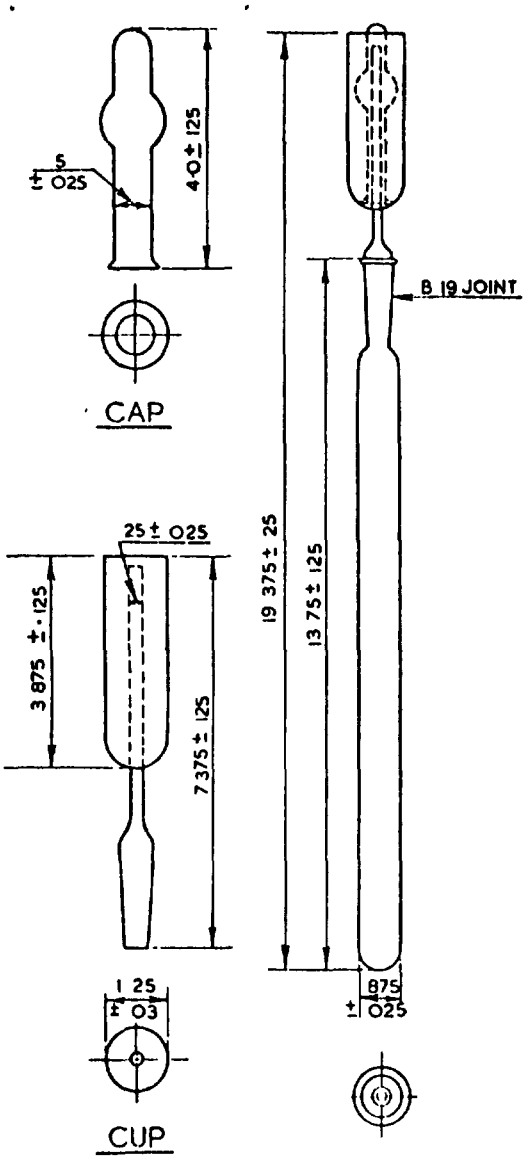
1. Fluorescent lamp, MCF, 460 mm tubular, 15 watt, colour matching, and suitable switch start gear.

2. Sheet aluminium reflector, to fit.

b. Alternatively, or additionally, overhead lighting may be used. The tube (80 watt, 150 cm in length, colour matching type) should be mounted in front of the bath so that its light is directed on to the top of the bath at an angle of about 45^{o}, from a distance of about one metre.

FIGURE 1 (Test 9)

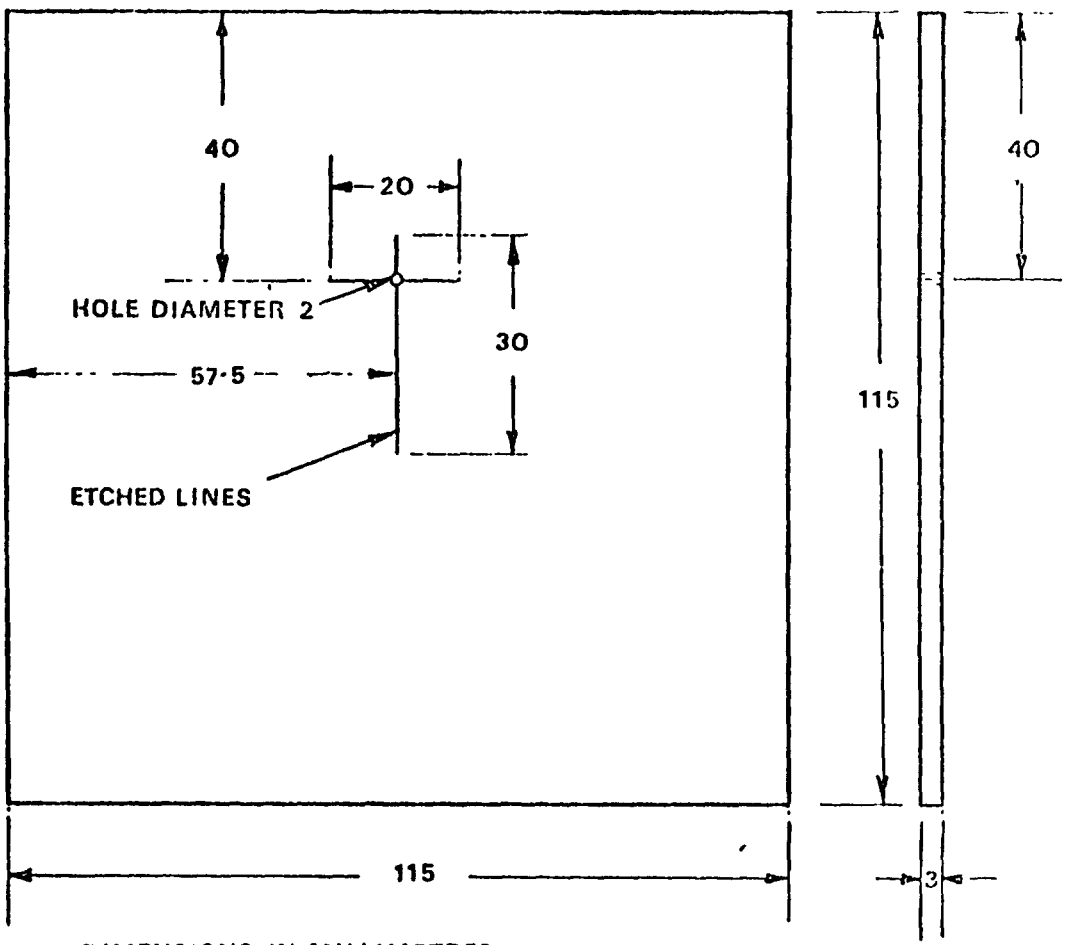
STABILITY TUBE
(132°C Stability Test) including Cap and Cup



TO BE CONSTRUCTED THROUGHOUT OF COLOURLESS 'RESISTANCE' GLASS. THE THICKNESS OF THE GLASS OF THE WALL OF THE STABILITY TUBE TO BE NOT LESS THAN 0.40 INCH (1.02 mm) NOR MORE THAN 0.050 INCH (1.27 mm).

FIGURE 2 (Test 10)

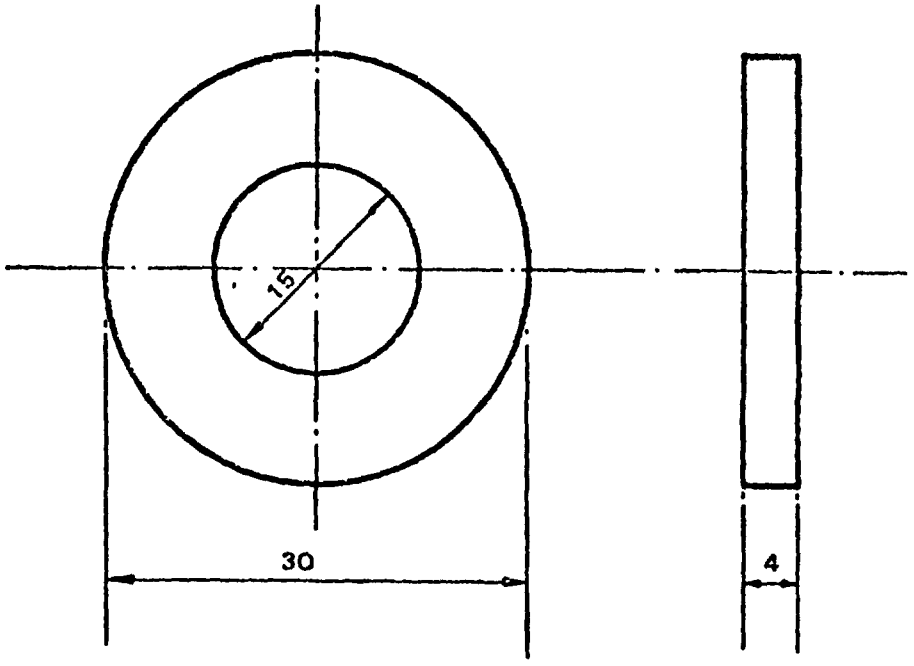
PLATE, GLASS, PERFORATED, MARK 1/C
(Apparatus Abel Heat Test)



DIMENSIONS IN MILLIMETRES

FIGURE 3 (Test 10)

RING, TEST TUBE, MARK 1/C
(Apparatus Abel Heat Test)

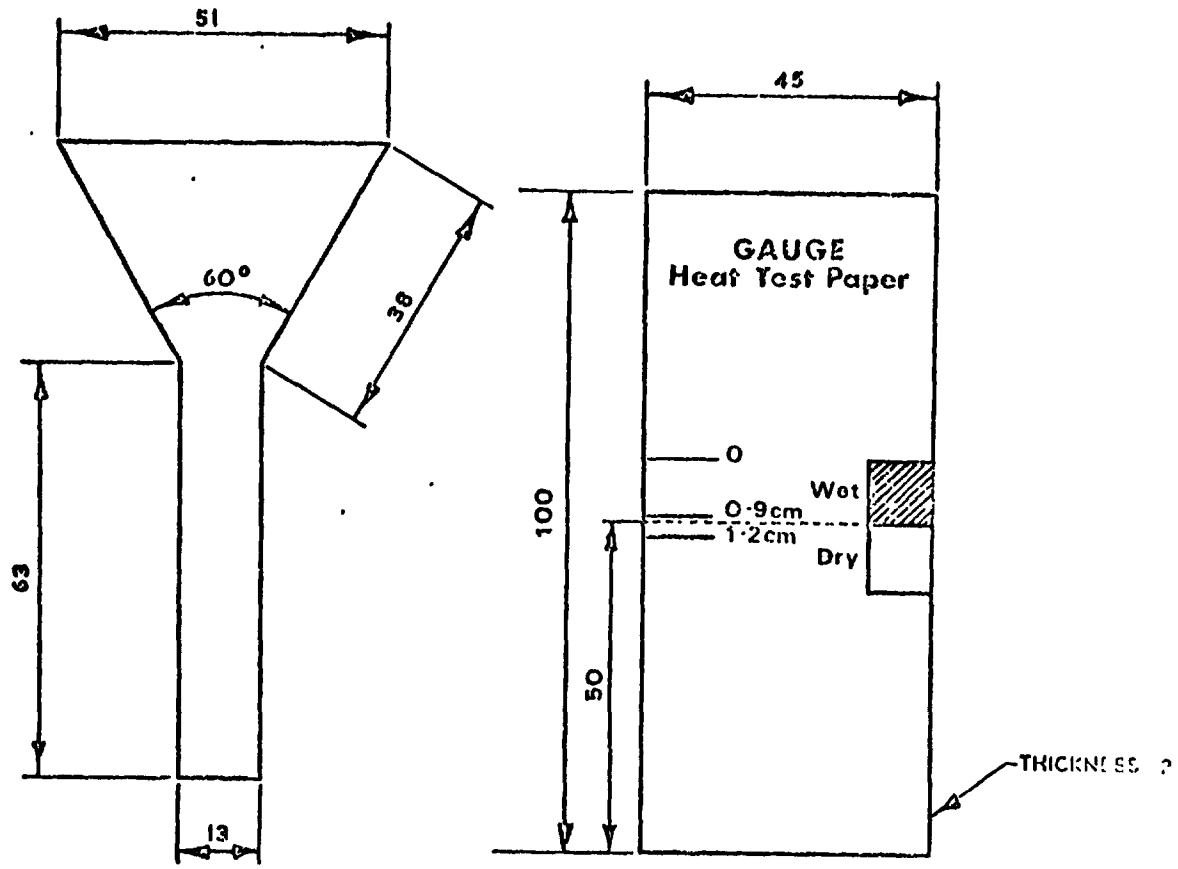


DIMENSIONS IN MILLIMETRES

ANNEX A to
STANAG 4178

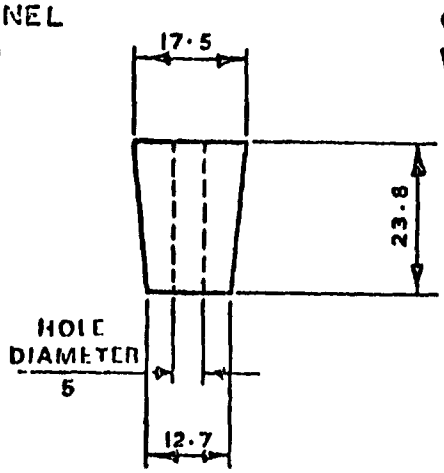
FIGURE 4 (Test 10)

FUNNEL, GAUGE AND STOPPER
(Apparatus Abel Heat Test)



ALUMINIUM FUNNEL
0.9 (20 SWG)

GAUGE
WHITE RIGID PLASTICS



RUBBER STOPPER FOR
HOOK ASSEMBLY

DIMENSIONS IN MILLIMETRES

11. EXAMINATION FOR THE PRESENCE OF STABILISING AGENTS

11.1 Outline of method

The nitrocellulose is extracted with di-ethyl ether in a Soxhlet apparatus. The extract, after concentration, is examined for the presence of stabilising agents by Thin Layer Chromatography.

11.2 Apparatus

Desaga chromatographic tank.
Thin layer chromatographic plates 20 x 20 cm coated with F254 (Merck) silica gel.
Micropipette.
UV lamp with dual sources, 254 nm and 366 nm.
Soxhlet extraction apparatus.
Water bath.

11.3 Reagents

Benzene pure.
Carbon tetrachloride, pure.
1:2 dichloroethane pure.
Di-ethyl ether, reagent grade, non-peroxide containing ethyl alcohol, reagent grade, non-denatured.

11.4 Procedure

Transfer 10 g of dried nitrocellulose to each of two Soxhlet extraction apparatuses and extract with di-ethyl ether for 24 hours on a boiling water bath. Remove the Soxhlet apparatuses from the bath and allow to cool, combine the extracts and evaporate to a small volume, dilute to approximately 10 cm^3 with ethyl alcohol and re-evaporate down to a total volume of 1 cm^3. Apply 10 µl of the ethyl alcohol concentrate by means of micropipette to a silica gel coated thin layer chromatographic plate which has been previously activated at 110°C. Allow the TLC plate to dry and develop in a solvent mixture comprising of:

5 parts benzene by volume
3 parts carbon tetrachloride by volume
2 parts 1:2 dichloroethane by volume

or suitable alternative contained in a chromatographic development tank. Remove the TLC plate when the solvent front has advanced 12 cm and allow to dry at room temperature. Examine the TLC plate under daylight conditions and with the aid of a UV lamp at 254 nm and 366 nm for the presence of spots, record the Rf value of any spots found. Identify the material present in the spot and confirm by comparison with standard reference substances.

11.5 Reporting

Report the absence or presence and identity of stabilising agents.

ANNEX B to
STANAG 4178
(Edition 1)

TEST REPORT SHEET

1. Laboratory: Country: Date:

2. Identification of test material.
Trade Name and/or Identity Code.
Manufacturer.
Lot, Batch or Consignment Number.
Date of Manufacture or Receipt.
Special Storage Condition.

3. Test Material Specification.

4. Test Results:

(i)	Mineral matter (state if carbonated)	%
(ii)	Grit retained No. 30 BS sieve Grit retained No. 60 BS sieve	particles particles
(iii)	Organic matter insoluble in acetone	%
(iv)	Organic matter soluble in ether/alcohol mixture	%
(v)	Fibre size/length	cm^3
(vi)	Viscosity	cps
(vii)	Nitrogen content (state method used)	%
(viii)	Stability at 132°C	mgm N_2/gm
(ix)	Heat Test at °C	minutes
(x)	Stabilising agents (with identification if found present).	

5. Divergences from Standard Procedures (if applicable).

RATIFICATION AND IMPLEMENTATION DETAILS
STADE DE RATIFICATION ET DE MISE EN APPLICATION

NATION	NATIONAL RATIFICATION REFERENCE DE LA RATIFICATION NATIONALE	NATIONAL IMPLEMENTING DOCUMENT NATIONAL DE MISE EN APPLICATION	IMPLEMENTATION/MISE EN APPLICATION					
			FORECAST DATE / DATE PREVUE			ACTUAL DATE / DATE REELLE		
			NAVY / MER	ARMY / TERRE	AIR	NAVY / MER	ARMY / TERRE	AIR
BE								
CA	2441-4178 (DAME 2) of/du 10.7.86	D-01-060-001/ AX-000		2.92				
DA	M.204.69-S 4178/MAS ARMY-26823 of/du 31.10.86							
FR								
GE	BMVg Fu S IV 1 Az 03-51-60 of/du 6.8.86		12.91	12.91	12.91			
GR								
IT								
LU								
NL	M86/0310/8468 of/du 30.7.86		8.92	8.92	8.92			
NO								
PO *	RRN 080/86/DD of/du 22.7.86		8.92	8.92				
SP								
TU								
UK	D/D Stan/341/8/4178 of/du 28.4.86	Def.Std	2.92	2.92	2.92			
US	Ltr AMSAC-MC/S of/du 3.9.87	MIL-STD-286 MIL-STD-244A				8.91	8.91	8.91

*See reservation overleaf/
Voir réserve au verso

RESERVATIONS/RESERVES

PORTUGAL

The tests numbers 7 (Viscosity) and 8 (Determination of nitrogen content) will not be applied.

Les essais numéros 7 (Viscosité) et 8 (Dosage de la teneur en azote) ne seront pas mis en application.